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High-Temperature Ceramic Coatings For Molybdenum

The lack of materials with the required properties for operating temperatures in excess of 1,800° F has been a restrictive influence in the development of ramjets, pulse-jets, turbo-jets, and rockets. The availability of materials suitable for use at higher temperatures than those in current use would effectively lessen design problems, making possible, for example, increased specific power in turbo-jet engines. Because the requirements of stress and temperature for such increased performance are beyond the potentialities of the currently used high-temperature alloys, scientific investigations at the National Bureau of Standards, under the sponsorship of the National Advisory Committee for Aeronautics, have been directed toward the development of ceramic coatings for the metals having very high melting points, in order to protect them against oxidation at high operating temperatures. Preliminary results indicate that molybdenum with a specially designed ceramic coating offers a promising combination for very high-temperature service.[1]

Most of the usual heat-resistant alloys begin to melt when heated within the temperature range 2,400° to 2,600° F. Of the metals whose melting points greatly exceed this temperature range, only such scarce metals as platinum and iridium, melting at 3,180° and 4,260° F respectively, have sufficient resistance to oxidation at high temperatures to be used without protection. Aside from the question of physical properties, the cost of these metals and the limited supply make their use on a large scale prohibitive.

Other metals that have high melting points, but lack adequate resistance to oxidation, include titanium (3,270° F), thorium (3,350° F), zirconium (3,450° F), boron (4,150° F), molybdenum (4,750° F), tantalum (5,160° F), and tungsten (6,100° F). The last three are of especial interest from the standpoint of potential applications in very high-temperature service. From a practical viewpoint, however, only molybdenum is available in very substantial quantities, there being comparatively large ore deposits in the United States. The preliminary work on the development of a protective ceramic coating that would permit metals of this type to be used at high temperature in oxygen-bearing atmospheres was therefore done with molybdenum.

The ceramic coatings developed to provide this protection to molybdenum are applied in the form of water suspensions, or "slips," to cleaned specimens of molybdenum by either dipping or spraying. After drying, the pieces are fired at a temperature of 2,150° F in oxygen-free atmospheres. The furnace is the only special equipment needed beyond that normally used in applying ceramic coatings.

[1] For further technical details, see NACA Technical Note 1626, copies of which are available from the National Advisory Committee for Aeronautics, 1724 F Street NW., Washington 25, D. C.

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A number of coatings were prepared. Some of these were outstanding in resistance to thermal shock, whereas others had good resistance to high temperatures. One of the better coatings, M–13–33, consisted of (1) a base coat of a low-expansion frit with 20-percent zirconia added, (2) a cover coat containing 95-percent zirconia, and (3) a seal coat consisting of a thin application of the same composition as the base coat.

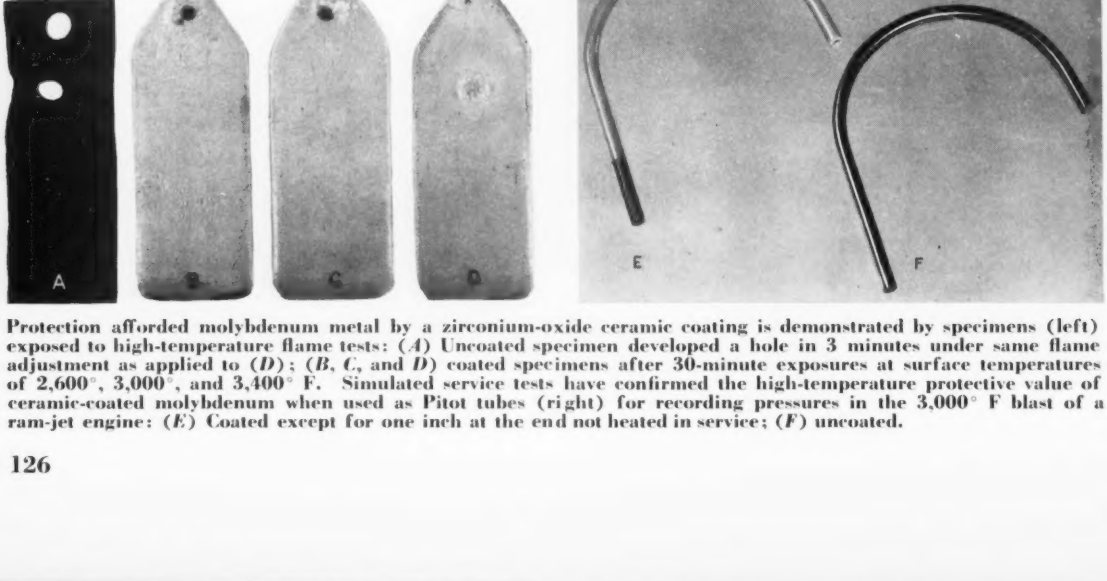
Coated specimens were subjected to several significant performance tests during the Bureau's investigations. These included heating in a gas-oxygen flame, heating at constant temperature in an air atmosphere, and service testing in the blast of ram-jet engines.

The results of these various tests indicated that the oxidation of the molybdenum was greatly retarded by the best of the ceramic coatings tried. In an air atmosphere at 1,650° F, unprotected molybdenum sheet was found to decrease 0.02 inch in thickness in ½ hour. There was no decrease, however, for ceramic-coated molybdenum heated for 70 hours under the same conditions.

At a gas temperature approximating 3,500° F, giving a surface temperature on the specimen of 2,600° F or more, only short-time protection of the molybdenum was attained. The oxidation rate at these high temperatures was found, however, to be sufficiently retarded by the presence of the ceramic coating to make the use of coated molybdenum feasible for special high-temperature applications in which prolonged service is not required.

An immediate application of these ceramic coatings is the protection of molybdenum Pitot tubes that are built into the nozzle end of ram-jet engines used for pilotless aircraft. These tubes, which are subjected to a gas temperature of about 3,000° F, are expendable and need not last over 5 minutes. A simulated service test of a ceramic-coated tube indicated a life in excess of 45 minutes.

The need remains for coatings which will have greater durability, and which can therefore be used for high-temperature applications where longer service life is required. However, in addition to Pitot tubes, other examples of equipment in which even short-time protection can be valuable include coated thermocouples and resistance thermometers made of high melting-point metals.



Protection afforded molybdenum metal by a zirconium-oxide ceramic coating is demonstrated by specimens (left) exposed to high-temperature flame tests: (*A*) Uncoated specimen developed a hole in 3 minutes under same flame adjustment as applied to (*D*); (*B*, *C*, and *D*) coated specimens after 30-minute exposures at surface temperatures of 2,600°, 3,000°, and 3,400° F. Simulated service tests have confirmed the high-temperature protective value of ceramic-coated molybdenum when used as Pitot tubes (right) for recording pressures in the 3,000° F blast of a ram-jet engine: (*E*) Coated except for one inch at the end not heated in service; (*F*) uncoated.

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Humidity Test Apparatus

A humidity test apparatus[2] developed and built in the mechanical instruments laboratory of the Bureau, now makes available an instrument for research, calibration, and testing of hygrometers at temperatures below freezing. The use in recent years of radiosondes for the meteorological sounding of the atmosphere, an important aid in weather forecasting, has created a marked need for information on the performance of the humidity-sensing element in the radiosonde under conditions of flight. The humidity test apparatus was developed under the sponsorship of the Bureau of Ships, Department of the Navy, to provide an instrument for the investigation of radiosonde hygrometer elements at the temperatures and air velocities encountered during a flight.

The function of the humidity test apparatus is to produce air of known relative humidity at temperatures from 0° to −40° C. In the new divided-flow, low-temperature humidity test equipment developed by Arnold Wexler, a current of dry air is divided into two streams, one of which is maintained dry while the other is saturated with respect to ice; finally the two are recombined.

A proportioning valve is used to divide the flow of moisture-free air in a known ratio. One part is passed through a saturator over a series of trays containing ice, until it is completely saturated. It is then mixed in a mixing chamber, with the other part that has been maintained dry, and allowed to exhaust through a test chamber into the atmosphere. The saturator, mixing chamber, and test chamber are kept immersed in a constant-temperature bath. The hygrometer or other device, which is being subjected to air of known and constant humidity, is inserted into the test chamber.

The relative humidity in the test chamber is a function of the fraction of air that passes through the saturator, the total pressure in the saturator, the total pressure in the test chamber, the saturation pressure, and the partial pressure of the water vapor in the test chamber. Under ideal conditions, which the apparatus closely approaches, all variables except one are eliminated, and the relative humidity becomes equal to the fraction of air that passes through the saturator.

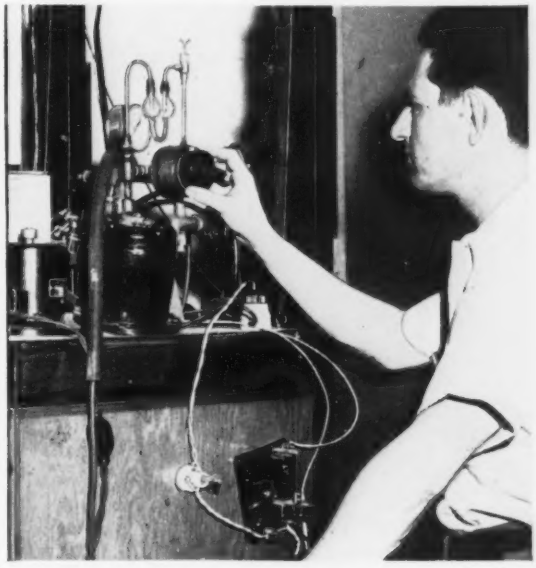
The essential functional units of the apparatus are the drying system, the proportioning system, the humidifying system, the mixing chamber, the test chamber, the cooling system, and the thermo-regulating system.

The drying system serves to remove all water from the air entering the apparatus by freezing in a dry ice-varsol bath. The dry air then passes through the proportioning system, the major feature of which is the proportioning valve. This valve, which divides the air in a definite ratio, consists of six orifices of equal cross-sectional area so arranged that by a turn of the knob of the valve the incoming air can be divided to produce any of seven ratios, 0, $\frac{1}{6}$, $\frac{1}{3}$, $\frac{1}{2}$, $\frac{2}{3}$, $\frac{5}{6}$, and 1. The ratio is the fraction of air entering the valve that emerges through one exit channel.

The two air streams, upon leaving the proportioning system, flow through the humidifying system in parallel channels, thermally in contact with one another to allow heat interchange and temperature equilibrium between the two streams. Saturation of one stream is accomplished by allowing it to pass over a series of staggered ice-filled trays, while the other stream passes over an identical path of dry shelves. The dry and saturated air are centrifugally mixed in a mixing chamber and discharged into the atmosphere through a $\frac{1}{8}$-inch inside diameter tube that serves as a test chamber. The saturators, mixing chamber and test chamber are immersed in a bath whose temperature is closely regulated.

The over-all performance of the humidity test apparatus has been established by a series of gravimetric moisture determinations. For the temperature range of 0° to −40° C and for air velocities up to 1,500 ft/min, this instrument produces air whose relative humidity is known within 3 percent.

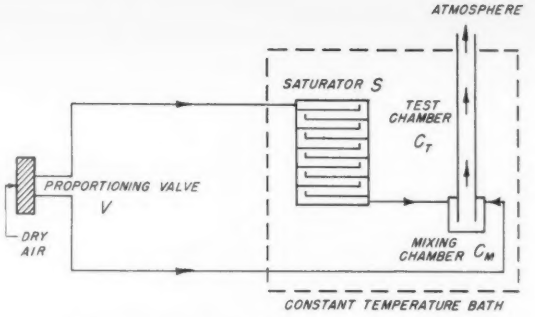
Although particularly suited for the investigation and calibration of hygrometers, psychrometers, and development apparatus at temperatures below 0° C, the humidity test equipment may find further application in general research and testing where air of a known low dewpoint or moisture content is desired. With such apparatus, devices placed in the test chamber can be subjected to discrete and almost instantaneous changes



The proportioning valve of the Bureau's new humidity test apparatus is so constructed that by a turn of the knob, the incoming air can be divided to produce any of seven ratios: 0, 1/6, 1/3, 1/2, 2/3, 5/6, and 1.

[2] For further technical details, see Divided flow, low-temperature humidity test apparatus, by Arnold Wexler, J. Research NBS 40, 479 (1948) RP1894.

Air of accurately known relative humidity is produced at temperatures below freezing according to the following basic principle: (1) Accurate division of a stream of dry air into two parts (V); (2) saturation of one part with respect to ice (S); and (3) recombining and mixing the two parts (C_M).



in relative humidity. Lag studies may therefore be easily made. The velocity of the air passing through the test chamber can be adjusted to any value up to 1,500 ft/min.

Although this instrument was designed primarily to fill a basic need in meteorology, the refrigeration and air conditioning industries may also find it useful. In all these fields, humidity plays an important role. With the humidity test apparatus, it is now possible to calibrate readily and accurately the various humidity measuring instruments employed at low temperatures.

Magnetic Measurement of Composite Copper-Nickel Coatings

A convenient, nondestructive magnetic method for determining the thickness of composite copper-nickel coatings electrodeposited on steel [3] has been developed by Abner Brenner and Eugenia Kellogg of the Bureau's electrodeposition laboratory. The method involves the measurement of the attractive force between the plated specimen and two permanent magnets of different strengths. The values thus obtained are used, in conjunction with a set of previously determined calibration curves for each magnet, to obtain the total thickness of the coating and the relative thicknesses of the copper and nickel layers.

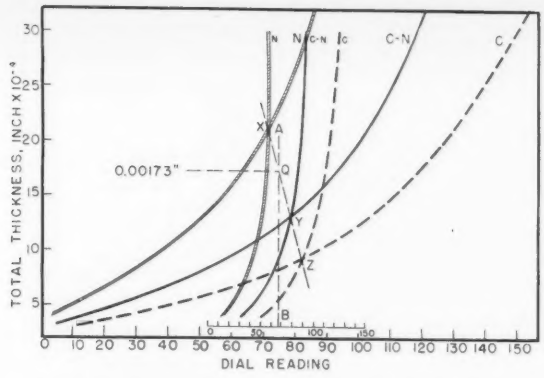
[3] For further technical details, see Magnetic measurement of the thickness of composite copper and nickel coatings on steel, by Abner Brenner and Eugenia Kellogg, J. Research NBS **40**, 295 (1948) RP1875.

Thickness of composite copper-nickel coatings electrodeposited on steel is determined by the Magne-gage, which indicates the force exerted by a spring in detaching a small permanent magnet from the plated sample.

Composite coatings, in which nickel is deposited over a layer of copper, are used extensively in the automotive and other industries for the economical protection of steel against corrosion. Although the effectiveness of these coatings is usually somewhat inferior to that of pure nickel plate, the copper required is less expensive than the extra layer of nickel that would otherwise be necessary. As the protective value of the composite coatings depends largely on their thickness, it is important to have a convenient means for measuring this property. In the usual procedure, the plated article must be destroyed to provide a cross section for microscopic examination. The new magnetic method, on the other hand, eliminates the necessity of cutting the specimen and is easily and rapidly applied.

This method utilizes the principle of the Magne-gage, an instrument originally designed at the Bureau to measure the thickness of single electrodeposited coatings on the basis of the attraction between a small permanent magnet and the plated sample. The Magne-gage is essentially a spring balance, on the arm of which a magnet is suspended in contact with the coated surface. A helical spring is so attached that, when wound by means of a knob, it exerts a force tending to detach the magnet from the surface. If, as is ordinarily the case, the coating is less magnetic than the object plated, the required force, as indicated on a dial adjacent to the knob, will be greater for thinner coatings.

For measurement of composite coatings, the Magne-gage is modified by the use of two magnets of different strengths. A set of calibration curves for each magnet—total thickness versus dial readings—is plotted on transparent material for coatings of successively varying known proportions of copper and nickel. After readings are taken with each magnet on the composite coating under study, one set of calibration curves is superimposed on the other in such a way that the values of the two separate dial readings on the horizontal



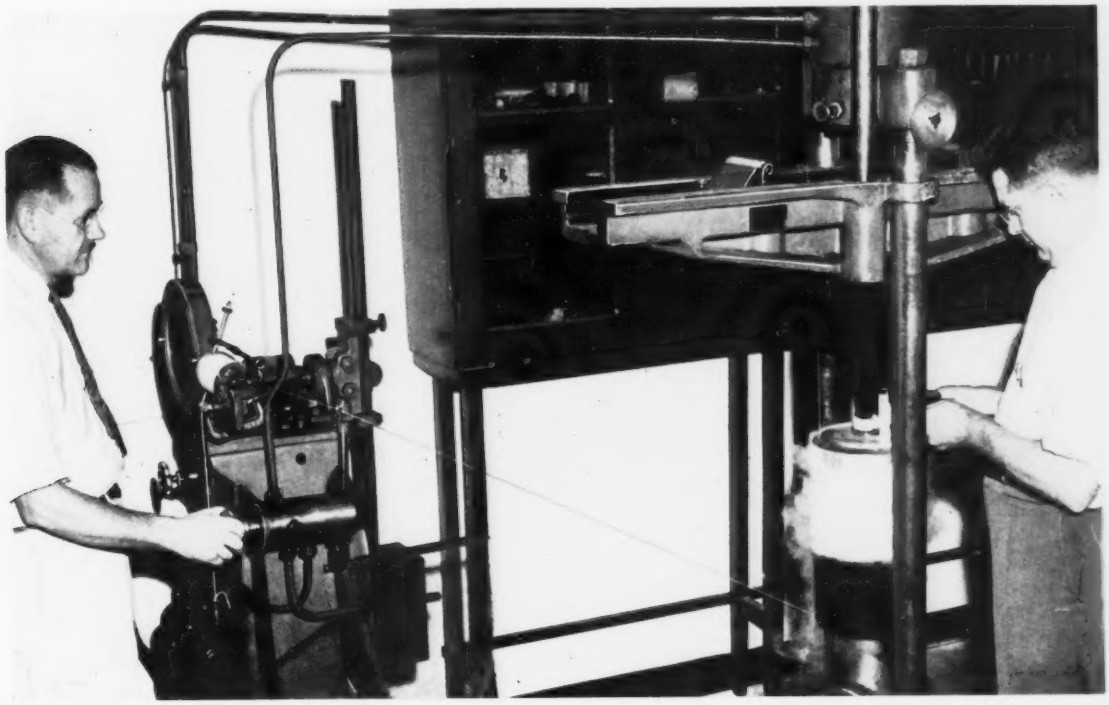
Two sets of calibration curves (for magnets of different strengths) form the basis of a graphical method for use with the Magne-gage in determining the thickness of composite copper-nickel electrodeposits. Superpose curves so that dial readings for each magnet coincide (*B*). Draw line *XYZ* through intersections of composition curves. Draw line *AB* perpendicular to horizontal axes to intersect *XYZ* at *Q*. Ordinate *Q* is thickness of total coating. Relative thicknesses of copper and nickel layers may also be obtained by graphical interpolation.

scale for each magnet are brought into coincidence. A straight line is drawn joining points of intersection of curves of the same percentage composition. If a second line is then drawn, perpendicular to the horizontal scales of the graphs, through the point representing the dial readings, it will intersect the first line in a point corresponding to the thickness of the total coating. The relative thicknesses of the copper and nickel layers are also obtained by graphical interpolation along the line joining the intersection points of the curves of equal composition.

In this way, total thicknesses of composite coatings ranging from 0.0005 to 0.003 inch can be determined within about 10 percent. The thickness of each component layer of similar coatings can be measured to an accuracy of about 15 percent.

Low-Temperature Properties of 18:8 Steel

For a number of years the Bureau has been engaged in a general research program concerning the influence of temperature on the mechanical properties of metals and alloys. One phase of the program deals with the



Mechanical properties of 18: 8 chromium-nickel steels at low temperatures have been determined by means of tension tests with circumferentially notched and unnotched cylindrical specimens, by using a pendulum-hydraulic testing machine. Tension tests at low temperatures are conducted with the steel submerged in a refrigerant bath (right) which may be maintained at temperatures as low as —188° C.

mechanical properties of austenitic 18:8 chromium-nickel steels at low temperatures ranging down to −188° C. Such information not only adds to fundamental knowledge of these materials but is also useful in predicting their behavior when subjected to the severe conditions encountered in high-altitude flying, arctic regions, and specialized refrigeration applications. Heretofore, information on the low-temperature properties of these steels has been meager, past investigations having been confined mainly to studies at room and elevated temperatures. In fact, the literature reveals very little, if any, data on the flow, fracture, and ductility characteristics of these materials at low temperatures.

Steels of 18:8 chromium-nickel composition, both with and without additions of molybdenum, titanium, and columbium, were included in the Bureau's investigation, which involved tests of the steels in annealed and cold-drawn conditions. When such steels are annealed, that is, cooled rapidly from about 1,900° F, they are austenitic. If they are cold-worked, however, some of the austenite transforms to ferrite containing carbon in super-saturated solution. Although the metastable condition of these steels is undesirable for certain applications, their remarkable strength and toughness make them suitable for many types of service, especially when high resistance to corrosion and oxidation is required. A ferritic 18:8 steel of the precipitation-hardening type was also included in the study.

The mechanical properties of the steels were determined by tension tests with circumferentially notched and unnotched cylindrical specimens by using a pendulum-hydraulic testing machine of 50,000 lb capacity. Tests at low temperatures were conducted with the specimens submerged in a refrigerant bath maintained at the selected temperature. In the special apparatus devised for these tests, the lower threaded end of a specimen extends through the bottom of a cylindrical-shaped vessel, containing the refrigerant, into the lower adapter of the testing machine. Lead washers are used to seal against loss of the bath liquid.

To obtain a temperature between room temperature and −78° C, a mixture of equal parts by volume of carbon tetrachloride and chloroform is cooled by pulverized solid carbon dioxide. Temperatures between −78° and −188° C are obtained with liquid dichlorodifluoromethane (CCl_2F_2) (Freon 12), which is cooled by pouring liquid air into the end of an immersed copper coil while the refrigerant is being constantly stirred. A chromel-alumel thermocouple attached to the specimen permits accurate control at the desired temperature. For a temperature of −188° C, the specimen is fully submerged in a bath of liquid air.

In these investigations the tensile load was applied more slowly than is usual in a tension test to avoid local heating of the specimen during the plastic deformation. The duration of the test was generally about 1 hour. Simultaneous load and diameter measurements were obtained throughout the duration of the tests at room temperature, and the flow-stress curves were derived directly from these measurements. Since diameter

Interference Fringes of Ne Merc

The wavelength of the green radiation of the artificial ry isoto intensity that can be obtained from any atom, either r or arti bombarding gold with neutrons in an atomic pile (N hnical pare the interference fringes formed by the green r of m seven-isotope natural mercury (left). From these fri he wav William F. Meggers of the Bureau's spectroscopy lab with tive to the wavelength of red radiation from cadmium ause o ience of the wavelength of mercury 198, the Natio eau of Conference of Weights and Measures for considerati e world

es of Mercury and Isotope 198

tificial ry isotope 198 represents the ultimate in sharpness and either n or artificial. Mercury 198 is a stable isotope made by pile (Nchnical News Bulletin 32, 133, December 1947). Comgreen ra of mercury 198 (right) with the complex fringes of hese fri the wavelength of mercury 198 has been measured by Dr. opy lab with an accuracy of one part in a hundred million relaadmium cause of the great precision, reproducibility, and conven- Nation eau of Standards is presenting it to the International ideratio e world's primary standard of length.

measurements could not be obtained during the tests at low temperatures, automatic load-extension diagrams were obtained from the head movement of the tensile machine. The flow-stress curves were derived from these diagrams.

Plastic deformation of the metastable austenitic steels during the tension test causes some of the austenite to transform to ferrite containing carbon in supersaturated solution. The rapidity of this phase change, which hardens and strengthens the steel, increases as the temperature is lowered.

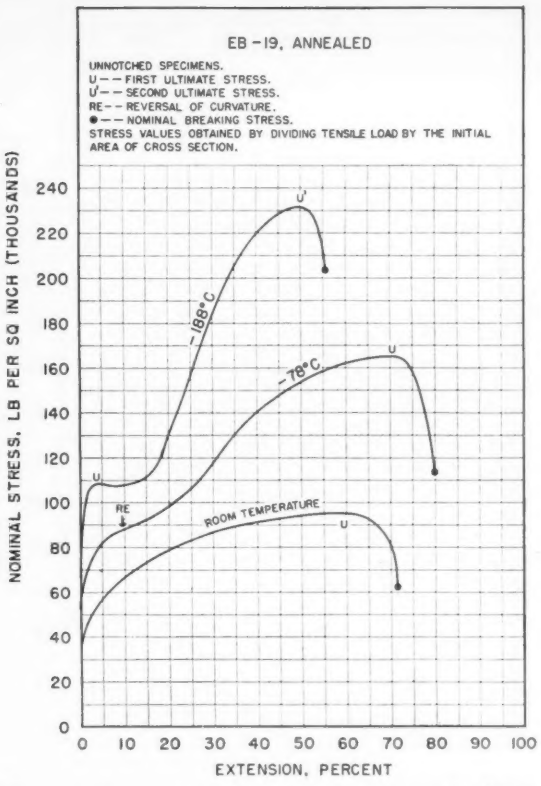
The automatic load-extension diagrams from the tension tests give clear evidence of phase change during the plastic deformation. The curves plotted from data taken in tests at room temperature are qualitatively similar to those obtained with a metal that does not undergo phase changes with plastic deformation. The slope of the curve decreases continuously between the points representing yield and maximum load. Transformation from austenite to ferrite during plastic deformation in the tension test at room temperature is not sufficient to cause a qualitative change in the form of the curves. The load-extension diagrams at $-78°$ and $-188°$ C, however, give evidence of a rapid phase change at these low temperatures. Some curves plotted from test data at $-78°$ C were found to exhibit a reversal of curvature with a rapid increase in slope beyond the point of reversal, showing that the strengthening during plastic deformation is due, not only to the ordinary work-hardening, but also to hardening caused by the phase change.

Other load-extension curves at $-78°$ C and curves at $-188°$ C clearly reveal still more evidence of the phase change. From the region representing the beginning of plastic strain, the curves rise with continuously decreasing slope to a maximum. The descent beyond the maximum represents the effect of incipient local contraction. However, the local contraction and consequent descent is stopped by a rapid phase change. The influence of the strengthening due to this phase change then becomes dominant causing the curves to rise to a second maximum. This second maximum point represents the beginning of final local contraction, and the curves descend to the point representing fracture of the specimens.

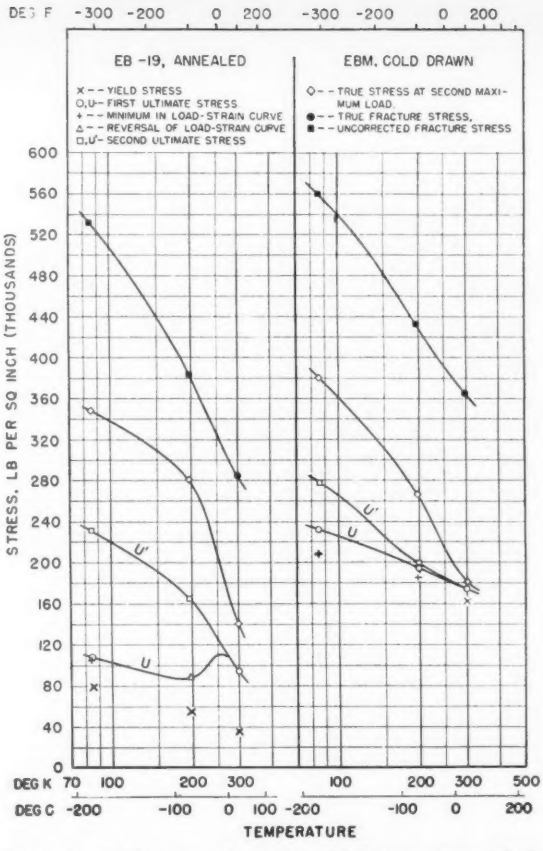
These investigations therefore show that six strength indices are sometimes required to evaluate the mechanical properties of these steels. These indices are: yield stress, first ultimate stress, stress at a reversal in the load-extension curve, stress at a minimum in the load-extension curve, second ultimate stress, and the true fracture stress.

The hardening and strengthening of 18:8 steels at low temperatures depends greatly upon the amount of prior plastic deformation (cold-working) the steel had received at room temperature. The increase in strength is greatest for the annealed steel and falls off with increase in the amount of prior plastic deformation at room temperature.

The metastable austenitic steels have good ductility. Even at temperatures as low as that of liquid air, the ductility, as determined by reduction of area, is about


EB -19, ANNEALED
UNNOTCHED SPECIMENS.
U - - FIRST ULTIMATE STRESS.
U' - - SECOND ULTIMATE STRESS.
RE - - REVERSAL OF CURVATURE.
● - - NOMINAL BREAKING STRESS.
STRESS VALUES OBTAINED BY DIVIDING TENSILE LOAD BY THE INITIAL AREA OF CROSS SECTION.
NOMINAL STRESS, LB PER SQ INCH (THOUSANDS)
EXTENSION, PERCENT
-188° C
-78° C
ROOM TEMPERATURE
U
U'
RE

Stress-strain curves reveal the behavior of annealed 18: 8 chromium-nickel steel at low temperatures. Note reversal of curvature (*RE*) during extension at −78° C., indicating a rapid phase change and the marked increase in slope beyond this point. Strengthening of the steel by rapid phase change during extension at −188° C. was so dominant that the curve shows two maxima.


EB -19, ANNEALED
EBM, COLD DRAWN
×- - YIELD STRESS
O,U- FIRST ULTIMATE STRESS
+ - - MINIMUM IN LOAD-STRAIN CURVE
△- - REVERSAL OF LOAD-STRAIN CURVE
□,U'- SECOND ULTIMATE STRESS
◇- - TRUE STRESS AT SECOND MAXIMUM LOAD.
●- - TRUE FRACTURE STRESS,
■- - UNCORRECTED FRACTURE STRESS
STRESS, LB PER SQ INCH (THOUSANDS)
DEG F
DEG K
DEG C
TEMPERATURE

Variation of the strength of plain 18:8 chromium-nickel steel with temperature. Curves of the variation of the second ultimate stress (*U'*) are affected by the phase changes induced by plastic deformation.

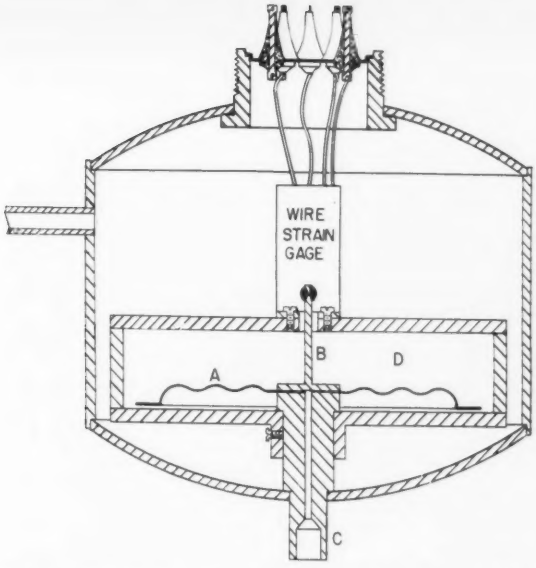
as great as at room temperature. In this respect they are similar to nonferrous metals having a face-centered cubic structure, which retain good ductility at low temperatures. The ductility of the metastable austenitic steels that have been previously subjected to plastic deformation is affected less by temperature than that of the annealed steels, as the amount of austenite transforming to ferrite in the tests at low temperature is less.

For the ferritic precipitation-hardening 18:8 steel, ductility decreases rapidly with decrease in temperature. At liquid-air temperature the steel is brittle. This 18:8 steel is similar therefore to the other ferritic steels, which lose ductility at low temperature and generally become brittle in tension tests at liquid-air temperatures.

A Highly Sensitive Differential Manometer

A differential manometer that will detect pressure differences as small as one millionth of an atmosphere over a range of total pressure from 1 mm of mercury to 1 atmosphere has been developed by Harry Matheson and Murray Eden of the Bureau's physical chemistry laboratories. This instrument, which operates on the principle of the aneroid barometer, can be used to compare pressures of gases, organic vapors, and noncorrosive liquids. It is particularly useful in vapor-pressure work, since less than a drop of liquid is sufficient to compare an unknown vapor pressure as a function of temperature with that of a known liquid. The rest point of the new manometer is insensitive to temperature, and accidental pressure differences as high as an atmosphere introduce only temporary hysteresis effects. The instrument is also readily portable and need not be leveled before operation.

A number of devices for measurement of absolute vacuum or pressure differences have been available to the laboratory worker. However, the majority of such



A new differential manometer measures very small differences in pressure of gases, organic vapors, and liquids. Changes in position of two nesting diaphragms, *A*, soldered at their edges to form a very thin capsule, are transmitted by means of rod *B* to a wire-resistance displacement gage (center of container, right). One substance is admitted to the interior of the capsule through *C*; the other surrounds the capsule in vacuum-tight compartment *D*. A galvanometer connected to the gage indicates the pressure difference.

gages do not operate satisfactorily in the presence of vapors. Other common disadvantages are large dead volume, inconvenience of handling or moving, difficulty or slowness of manipulation, lack of precision, and sensitivity to temperature. In connection with a study at the Bureau of high-polymer plastics in solution, it became necessary to compare solution vapor pressures accurately. Lack of suitable apparatus for this purpose resulted in the design of the new type of differential manometer, which is expected to prove useful in other fields of research. It may also be advantageously substituted for slope gages and other commercial devices for the measurement of small differences of pressure.

The instrument developed at the Bureau measures a pressure difference as the change in the separation of a pair of nesting diaphragms indicated by an unbonded wire-resistance displacement gage. The two diaphragms are soldered together at their edges to form a very thin capsule into which is admitted one of the vapors whose pressure is to be compared. The other vapor surrounds the capsule externally.

One of the diaphragms is attached to a stationary support through which vapor enters the capsule; the other is connected to the displacement gage. The four resistance wires of the gage are arranged to form a Wheatstone bridge, in which an increased separation of the diaphragms elongates one pair of wires and shortens the other pair. A decrease in the diaphragm spacing has the opposite effect. The resistive unbalance as indicated by a galvanometer gives an accurate measure of the separation of the diaphragms and thus of the pressure difference. The capsule and gage are sealed in a vacuum-tight chamber.

Tests of the new manometer have shown that its sensitivity is not appreciably altered when the active elements are used at temperatures between 20° and 50° C. for values of absolute pressure between 1 mm of mercury and 1 atmosphere. The elements can withstand a differential pressure of 1 atmosphere where the greater pressure is external to the capsule, or a pressure difference of 0.2 atmosphere in the other direction. The equality of two pressures may be found to 0.001 mm of mercury; the direction and magnitude of a pressure inequality may be determined between 0 and ± 0.5 mm of mercury. The indicated rest point remains constant within 0.005 mm of mercury for periods of at least 1 day, even though the device may be subjected during this time to pressure differentials of the order of 0.2 atmosphere and temperature variations of several degrees.

Seminar on High Polymers

The 1948–49 lectures on the chemical and physical properties of high polymers, given by the Nation's leading scientists in this field, continues the series of seminars that have been presented for the past 3 years at the Bureau. The program, arranged under the chairmanship of Dr. Robert Simha of the Division of Organic and Fibrous Materials, consists of seven lectures, which are open to the public without charge. The lectures this year, as indicated by the following titles, are of particular interest to research workers in rubber,

leather, and plastics as well as those concerned with the theory of high polymers.

October 7: Dielectric and mechanical properties at high frequencies, G. Gee, British Rubber Producers Research Association.

November 4: Some structural and chemical properties of collagen fibers, F. O. Schmitt, Massachusetts Institute of Technology.

January 6: Rheological properties of polystyrene, R. S. Spencer, Dow Chemical Company.

February 24: Some aspects of dynamic rubber-like elasticity, A. W. Nolle, University of Texas.

March 3: Reactions of free radicals with hydrocarbons, E. W. R. Steacie, National Research Council.

April 7: Variables which influence the properties of chemical rubbers prepared by emulsion polymerization, C. F. Fryling, Phillips Petroleum Company.

May 5: The chemistry of some derived polymers of the vinyl series, W. O. Kenyon, Eastman Kodak Company.

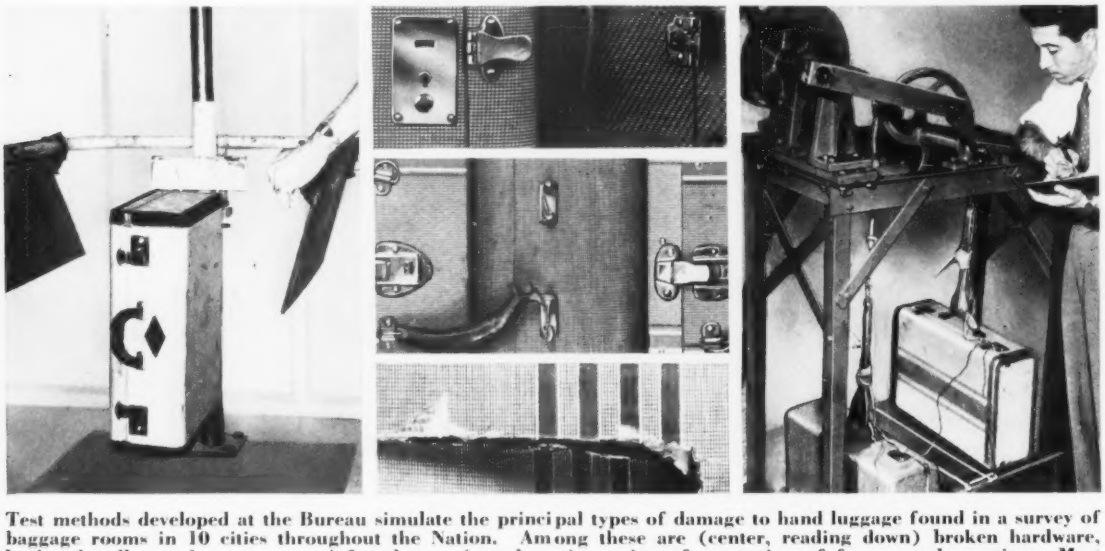
Evaluation of Hand Luggage Performance

Four new laboratory performance tests for evaluating the durability of hand luggage have been developed by the Bureau. The methods include a drop test, a static load test, a method for estimating the resistance to puncture, and a fatigue test for handles. These tests were devised in the course of research conducted to obtain sufficient technical data to permit promulgation of a commercial standard for hand luggage.

The seriousness of the problem is indicated by statistics that show that loss and damage to luggage in the hands of common carriers amounts to almost one and a quarter million dollars annually. The possibility of reducing such loss by better methods of handling and by the design and construction of sturdier luggage was suggested by rail, bus, and air traffic officials and, at their request, investigations were begun at the Bureau of Standards. The development of methods of testing bags by simple procedures, applicable for judging their durability, made it necessary to obtain preliminary information of a general and technical nature concerning luggage in actual service.

A survey of baggage rooms in 10 cities throughout the United States revealed that 64 percent of all checked luggage consisted of week-end and overnight cases (considered as one class), wardrobe cases, and suitcases. The most popular covering materials varied somewhat with locality, but in general, fabric-covered cases, the so-called "airplane" luggage, amounted to approximately 40 percent of the items surveyed, with leather and fiberboard rating next. Degree of loading (pounds per cubic foot) tended to decrease with increase in size. Based on the data collected, the loading for laboratory test purposes (including the weight of the bag) varied from 22 to 77 pounds. Principal types of damage found in the survey were broken hardware, broken handles, separation of frames or covering, punctured surface, scuffing and tearing, and soiling. The first four types may practically destroy the functional usefulness of the container, and therefore laboratory methods were directed toward duplicating such damage.

The most effective of the four tests devised by Bureau scientists was the drop test, which uses a divided table



Test methods developed at the Bureau simulate the principal types of damage to hand luggage found in a survey of baggage rooms in 10 cities throughout the Nation. Among these are (center, reading down) broken hardware, broken handles, and punctures. A fourth type (not shown) consists of separation of frames and covering. Most effective of the tests is a drop test (left), which uses a divided-table drop-tester. Handles are tested by subjecting loaded bags to action of a machine (right), which picks them up by the handle and sets them down repeatedly.

drop-tester meeting the requirements of ASTM Tentative Specification D775–45T. The loaded bags were dropped consecutively on each of their faces, edges, and corners from heights beginning at 1½ feet and increasing at 6-inch increments to a maximum of 5 feet. For each fall, scores were assigned commensurate with the damage occurring. Summation of the scores at completion of the tests or at failure gave a quantitative evaluation of the resistance of the bags to dropping.

Handles were tested by subjecting the loaded bags to the action of a machine that picks them up by the handle and sets them down repeatedly. The durability of the plate through which the handle is fastened to the bag, as well as of the handle itself, was thus tested. The apparatus is equipped with a cycle counter and a device for stopping its operation when failure of a handle occurs.

The ability of a bag to withstand static loads without suffering permanent deformation was tested by placing successively on side, top, and bottom a 150-pound weight for a period of 5 minutes in each position.

Puncture resistance of covering materials was determined on sections cut from the surfaces of the bags, by using an instrument primarily designed for testing the puncture resistance of fiberboard shipping containers. Puncture values in inch-ounces were read directly from a scale on the apparatus.

Preliminary tests demonstrated suitability of these methods for simulating actual service damage, and new bags, representing the most popular style and covering materials, were purchased in triplicate, in large and small models. Two of each set were tested by the laboratory procedures, and the third was placed in service, loaded as previously described and shipped by rail approximately 12,000 miles, on 22 round trips from Washington to predetermined points in the country, receiving 24 handlings per round trip. The damage was evaluated by a score system similar to that used for the drop test.

The results of the service tests and three of the laboratory tests (drop, handle fatigue, and static load) showed a strong correlation. Data from the test for puncture resistance were insufficient to permit final conclusions, although they indicated that the test is a valuable one. The samples with lowest puncture values were comparably damaged in service, but some bags with relatively high puncture resistance also received punctures in travel.

Minimum performance requirements tentatively recommended are that the piece of luggage shall withstand 150 pounds placed on top, side, and ends for 5 minutes in each position; shall withstand 25,000 pick-up motions of the handle-fatigue test apparatus; and shall obtain a specified score of quality (roughly equal to 50 drops) when dropped from various height levels on faces, edges, and corners.

Standard Samples for Rubber Compounding

As an outgrowth of the successful use of standard samples of rubber-compounding ingredients in the Government synthetic rubber program, the Bureau has included seven of these standards among the standard samples regularly supplied to industrial and scientific laboratories. The requirements of the specifications for Government synthetic rubbers are now predicated on the use of these standard samples for stress-strain testing of synthetic rubbers. The samples are also available to manufacturers of rubber products for use in connection with the procurement of raw materials, process control, and inspection of product quality.

The use of standard samples of compounding ingredients for the testing of rubber was inaugurated in 1943 by the Committee on Specifications for Synthetic Rubbers (sponsored by the Office of Rubber Director and Rubber Reserve Co., now Office of Rubber Reserve). This was a vital part of the Government synthetic rubber program, which resulted in outstanding improvements in precision and accuracy of testing in the Government synthetic rubber plants. The uniformity of synthetic rubber production in recent years has been made possible by the improvements in testing rubber.

Orders for these materials should be sent to the National Bureau of Standards, Washington 25, D. C., accompanied by payment in advance. Remittances should be payable to the National Bureau of Standards. Shipments will be made express collect, unless otherwise specified. The following materials are available in air-tight metal containers except Standard Sample No. 376, which is packaged in glass. (When ordering, specify *both number and name of sample.*)

Standard Sample No.	Name	Weight per package	Packages per carton	Price per package
		g		
370	Zinc oxide	2,000	8	$1.65
371	Sulfur	1,000	20	.60
372	Stearic acid	600	1	1.20
373	Benzothiazyl disulfide	600	40	1.20
374	Tetramethylthiuram disulfide.	600	1	2.25
375	Channel black	8,000	4	3.50
376	Light magnesia	110	1	.85

Standard Time Zone Map

A revised map of the standard time zones of the United States and adjacent parts of Canada and Mexico has been issued as Miscellaneous Publication M190 of the National Bureau of Standards. The time zone boundaries, based on the most recent rulings of the Interstate Commerce Commission, are shown as of January 1, 1948. This map is available only from the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C., at 15 cents a copy.

NBS Scientists

Dr. Newbern Smith has been designated Chief of the Central Radio Propagation Laboratory, succeeding **Dr. J. H. Dellinger,** whose retirement was announced recently. Dr. Smith will plan and direct basic theoretical and experimental research on radio wave propagation; operation of the world-wide network of radio propagation observatories; and development of measurement standards at all radio frequencies. During the war he served as assistant chief, supervising research, of the Interservice Radio Propagation Laboratory, set up by the U. S. Joint Chiefs of Staff to collect, analyze, and disseminate data on ionospheric radio propagation. This program was centralized in the radio propagation division of the Bureau after the war.

NBS Publications

Periodicals [1]

Journal of Research of the National Bureau of Standards, volume **41**, number 4, October 1948. (RP1922 to RP1929 inclusive.)

Technical News Bulletin, volume **32**, number 10, October 1948. 10 cents.

CRPL-D50. Basic Radio Propagation Predictions for January 1949. Three months in advance. Issued October 1948. 10 cents.

Nonperiodical

Research Papers [1,2]

RP1915. Thickness of inhibiting films on glass electrode surfaces. Donald Hubbard and Gerald F. Rynders. 10 cents.

RP1916. Light-sensitive papers as controls for testing textile colorfastness and stability of materials under arc-lamp exposure. Herbert Launer. 10 cents.

RP1917. Expansive characteristics of hydrated limes and the development of an autoclave test for soundness. Lansing S. Wells, Walter F. Clarke, and Ernest M. Levin. 15 cents.

RP1918. Concentration of isotopes of mercury in countercurrent molecular stills. Samuel L. Madorsky, Paul Bradt, and Sidney Straus. 10 cents.

RP1919. Determination of lactose alone and in the presence of sucrose by the method of Munson and Walker. Lester D. Hammond. 10 cents.

RP1920. Broad- and narrow-beam attenuation of 500- to 1,400-kilovolt X-rays in lead and concrete. Harold O. Wyckoff, Robert J. Kennedy, and William R. Bradford. 10 cents.

RP1921. A method for the electron miscroscopy of wool. Max Swerdlow and Gloria S. Seeman. 10 cents.

Miscellaneous [1]

M189. Report of the Thirty-third National Conference on Weights and Measures. 40 cents.

Simplified Practice Recommendations [1]

R233–48. Rotary files and burs. 10 cents.

R234–48. Welded-wire fabric reinforcement for concrete pipe. 5 cents.

R235–48. Copper and copper-alloy round seamless tube. 5 cents.

Letter Circulars [6]

LC915. Painting exterior walls of porous masonry. (Supersedes LC747.)

LC916. Sources of radio information. (Supersedes LC850).

LC917. Artificial abrasives and abrasive products. (Supersedes LC812.)

LC918. Leather: Publications by the staff of the National Bureau of Standards. (Supersedes LC788.)

LC919. List of weights and measures offices of the States and the District of Columbia. (Supersedes LC834.)

LC920. Equipment for the testing of large-capacity scales.

LC921. Leather gloves: General information.

Articles by Bureau Staff Members in Outside Publications [7]

Purification of uranium oxide. James I. Hoffman. J. Wash. Acad. Sci. (c/o Harold A. Rehder, U. S. National Museum, Washington 25, D. C.) **38**, 233 (1948)

A method for the electron microscopy of wool. Max Swerdlow and Gloria S. Seeman. Textile Research j. (10 East Fortieth Street, New York 16, N. Y.) **18**, 536 (1948)

Coefficients for complex quartic, quintic, and sextic interpolation within a square grid. Herbert E. Salzer. J. Math. and Phys. (Massachusetts Institute of Technology, Cambridge 39, Mass.) **27**, 136 (1948)

Tables of coefficients for interpolation in functions of two variables. H. E. Salzer. J. Math. and Phys. (Massachusetts Institute of Tech., Cambridge, Mass.) **26**, 294 (Jan. 1948)

Reinforcement of a small circular hole in a plane sheet under tension. Samuel Levy, A. E. McPherson, and Frank C. Smith. J. App. Mech. (American Society of Mechanical Engineers, 29 W. Thirty-ninth Street, New York 18, N. Y.) **15**, 160, (168)

Pyrolitic fractionation of polystyrene in a high vacuum and mass spectrometer analysis of some of the fractions. S. L. Madorsky and S. Straus. Ind. & Eng. Chem. (1155 Sixteenth Street, NW, Washington 5, D. C.) **40**, 848 (1948)

The aging of GR-S. A review of the literature. Thomas H. Harris and Robert D. Stiehler. India Rubber World (386 Fourth Avenue, New York 16, N. Y.) **113**, 365 (1948)

Strain test for evaluation of rubber compounds. Frank L. Roth and Robert D. Stiehler. India Rubber World **113**, 307 (1948)

Sulfur compounds in gasoline. Abraham Fookson and Albert D. Bell. Petroleum Refiner (Gulf Publishing Co., Box 2608, Houston, Tex.) **27**, No. 9, Sec. 1, 87 (Sept. 1948)

Coefficients for expressing the first twenty-four powers in terms of Legendre polynomials. H. E. Salzer. Mathematical Tables and Other Aids to Computation, (Brown Univ., Providence 12, R. I.) **3**, No. 21, 16 (1948)

Errors in calibration of the *f*-number. Francis E. Washer. J. Soc. Motion Picture Eng. (342 Madison Avenue, New York, 17, N. Y.) **51**, No. 3, 242 (1948)

Precision measurement of electrical characteristics of quartz-crystal units. W. D. George, M. C. Selby, and R. Scolnik. Proc. IRE (1 E. Seventy-ninth St., New York 21, N. Y.) **36**, 1122 (1948)

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[1] Send orders for publications under this heading only to the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Annual subscription rates: Journal of Research, $4.50 (foreign $5.50); Technical News Bulletin, $1 (foreign $1.35); Basic Radio Propagation Predictions, $1 (foreign $1.25). Single copy prices of publications are indicated in the lists.

[2] Reprints from September Journal of Research.

[6] Letter Circulars are prepared to answer specific inquiries addressed to the National Bureau of Standards, and are sent only on request to persons having a definite need for the information. The Bureau cannot undertake to supply lists or complete sets of Letter Circulars or send copies automatically as issued.

[7] These publications are not available from the Government. Requests should be sent direct to the publishers.

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